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Acta Materialia 51 (2003) 6253–6261
Acta MATERIALIA
www.actamat-journals.com

Tin whiskers studied by synchrotron radiation scanning X-ray micro-diffraction

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Received 3 May 2003; received in revised form 28 July 2003; accepted 29 July 2003

Abstract

A large number of Sn whiskers have been found on the Pb-free solder finish on leadframes used in consumer electronic products. Some of the whiskers on eutectic SnCu finishes are long enough to short the neighboring legs of the leadframe. Tin whisker growth is known to be a stress relief phenomenon. We have performed synchrotron radiation X-ray micro-diffraction analysis to measure the local stress level, the orientation of the grains in the finish around a whisker, and the growth direction of whiskers. The compressive stress in the solder finish is quite low, less than 10 MPa; nevertheless, there exists a stress gradient around the root of a whisker. From the orientation map and pole figure, we found that the growth direction of whiskers is [0 0 1] and there exists a preferred orientation of [3 2 1] grains on the solder finish. In one of the whisker analyzed, we found that the normal orientation of the grain just below the whisker is different; it is [2 1 0].

Keywords: Tin whisker; Pb-free solder; X-ray micro-diffractions; Stress gradient; Grain orientation

1. Introduction

Currently, Sn whisker growth is of interest owing to the application of Pb-free solder in electronic manufacturing [1]. The leadframe, which is used to interconnect a chip to its packaging board in large volume consumer electronic products, is electroplated or finished with a layer of Pb-free solder. The finish passivates the leadframe surface as well as enhances wetting in solder reaction. The Pb-free solder is typically pure Sn or eutectic SnCu (0.7 at.% Cu). On the SnCu finish, many long Sn whiskers, over 0.3 mm, have been found, as shown in Fig. 1. They are long enough to short the neighboring legs of the leadframe. Generally speaking, whisker and hillock growths are stress relief phenomena [2–8]. They relieve the compressive

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1359-6454/$30.00
doi:10.1016/S1359-6454(03)00448-8

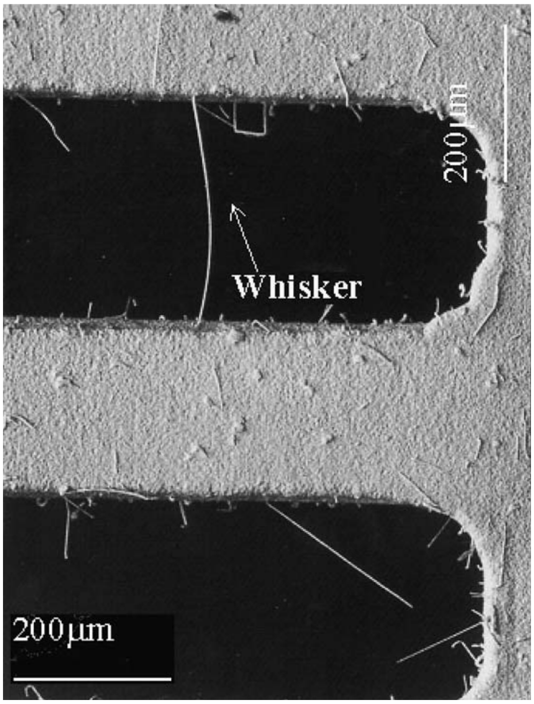

Fig. 1. Scanning electron microscopic (SEM) image of leadframe legs shorted by a Sn whisker.

stress in the matrix on which they grow. Spontaneous Sn whiskers are known to grow from the bottom [4], so they are pushed out by compression, as in making spaghetti. The mass transport is by atomic diffusion as in creep.

The origin of the compressive stress in whisker growth can be mechanical, thermal, and chemical in nature. The latter is dominant but less obvious. Spontaneous growth of Sn whiskers requires a driving force which endures with time. Both mechanical and thermal stresses are finite, so they cannot sustain the constant rate of growth of whiskers. The origin of the chemical force is due to the reaction between Sn (solder) and Cu (leadframe) to form the intermetallic compound (IMC) Cu_6Sn_5 at room temperature [9,10]. As long as the reaction proceeds at room temperature, the driving force is maintained.

Compressive stress has been found to be a necessary but insufficient condition for whisker growth. Another necessary condition is a protective surface oxide. This is because in ultra-high vacuum, the free surface of Sn becomes a good source and sink of vacancies, so the compressive stress can be relieved uniformly in each of the Sn grains, on the basis of the Nabarro–Herring model of creep. Thus, the relaxation is uniform and no localized growth of whiskers will take place [11]. For whisker growth, the surface cannot be oxide free. Furthermore, the oxide must be a protective one so that it removes all the vacancy sources and sinks on the surface effectively. Hence, only those metals which grow a protective oxide, such as Al and Sn, are known to have hillock or whisker growth. Metals with a non-protective oxide, such as Fe and Cu, will not grow hillocks and whiskers. On the other hand, if the surface oxide is very thick, it will physically block the protrusion of a hillock or whisker. Thus, the protective surface oxide should be broken at certain weak spots on the surface, from where the whiskers grow to relieve the stress [12,13]. Surrounding a broken spot, a lateral stress gradient is produced in the Sn matrix, and the gradient becomes the driving force for the long range atomic diffusion of Sn needed to grow the whisker. Since the room temperature chemical reaction maintains the stress and the oxide maintains the gradient, the whisker growth is spontaneous and localized.

We have performed synchrotron radiation X-ray micro-diffraction analysis to measure the compressive stress, stress gradient, grain orientation distribution in the finish, and growth direction of Sn whiskers.

2. Experimental

The X-ray micro-diffraction apparatus in the end-station (no. 7.3.3.) at Advanced Light Source (ALS) in Lawrence Berkeley National Laboratory was used to study Sn whiskers grown on the eutectic SnCu finish at room temperature. The ALS is capable of delivering a white X-ray beam (6–15 keV) focused to 0.8–1 μm via a pair of elliptically bent Kirkpatrick–Baez mirrors. In the apparatus, the beam can be step-scanned over an area of 100 μm × 100 μm at steps of 1 μm. Fig. 2 shows the schematic diagram of the geometry of the apparatus. Since the diameter of the whisker and

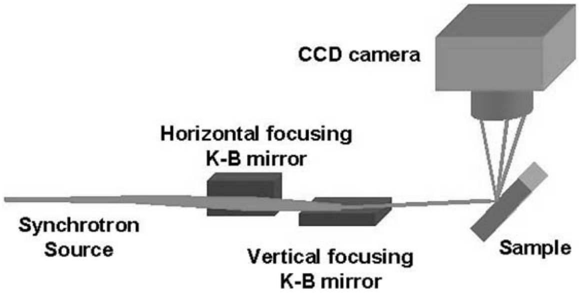

Fig. 2. Schematic diagram of the set-up for X-ray micro-beam diffraction analysis. The *X*-axis is perpendicular to the length of the leg, the *Y*-axis is parallel to the length of the leg, and the *Z*-axis (not shown) is parallel to the surface normal of the leg.

the size of grains in the finish are larger than 1 μm, each of them can be treated as a single crystal with respect to the micro-beam, so we obtain structural information such as stress and orientation by using white beam (Laue) diffraction. The technique of scanning X-ray micro-diffraction (μSXRD) that we used here has been described by Tamura et al. [14]. We have scanned several areas of the SnCu finish and we had chosen those areas in each of them where there was a whisker, especially the areas that contained the root of a whisker.

Laue patterns were collected with a large area (9×9 cm^2) charge coupled device (CCD) detector with an exposure time of 1 s per step of scan, from which the orientation and strain tensor of each illuminated grain can be deduced. Because of the low absorption of X-rays, several grains through the thickness of the finish are illuminated under the X-ray at the same time, but the grain of interest (the one just below the surface) can be discriminated from the rest by the intensity of reflections. One example of Laue pattern from a Sn grain is shown in Fig. 3. The crystal orientation and the lattice parameters of the Sn whisker and the grains of SnCu matrix surrounding the root of the whisker were measured by the Laue patterns. The software in ALS is capable of determining the orientation of each of the grains and displaying the distribution of the major axes of these grains. In addition, using the lattice parameters (Sn has a crystal structure of body-centered tetragonal with lattice parameters of $a = b = 0.58311$ nm and $c = 0.31817$ nm) of the whisker as stress-free internal reference, the strain or stress in the Sn grains in the SnCu matrix can be determined and displayed. The resolution of the white beam Laue technique is 0.005% strain. We recall that use of the Laue pattern is not a high precision method for lattice parameter determination. Hence, the internal stress-free reference (whisker) is useful and it enables us to determine the relative stress and stress gradient in the grains with respect to the whisker.

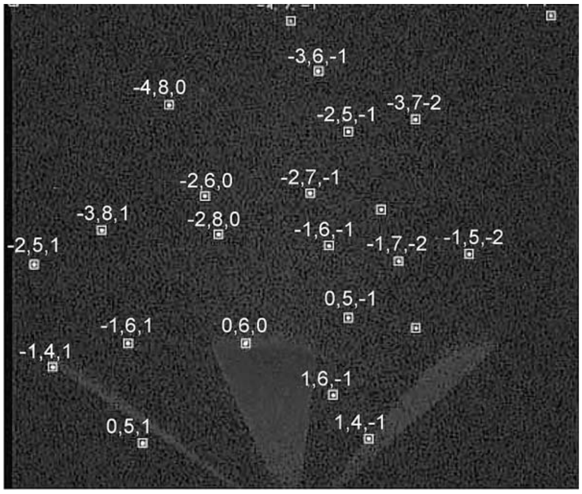

Fig. 3. X-ray micro-beam diffraction pattern (Laue pattern).

3. Results

3.1. Direction of whisker growth and orientation of the matrix

Fig. 4 shows a schematic diagram of an area of solder finish wherein the whisker scanned is indicated by the broken square. (A schematic diagram of the cross-sectional view of the whisker and solder finish will be presented later in Fig. 9.) This whisker is straight and its projection is almost perpendicular to the edge of the leadframe. The micro-beam scanned the root of the whisker and the surrounding grains of the whisker within an area of 100 μm × 100 μm. From the scan, information on the orientation of each scanned spot was obtained. The orientation map or the distribution of the angle between the normal vector (3 2 1) of each scanned spot of area of about 2 μm × 2 μm and the labora-

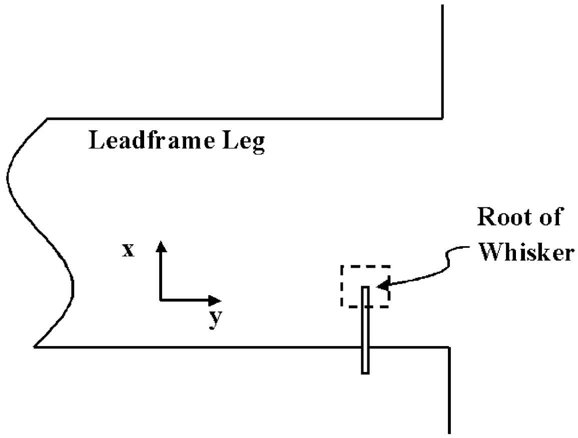

Fig. 4. Schematic diagram of a surface of leadframe which contains a whisker. The square indicates the area scanned by micro-beam.

tory Z-axis is shown in Fig. 5. The long bright line image in the middle is from the whisker. The arrow points to the root of the whisker (the end of the long bright line image), below which a [2 1 0] oriented grain was found. The analysis of the orientation of this grain, which is part of the whisker, will be presented later. We take Z to be the axis parallel to the surface normal, and X and Y to be the orthogonal axes in the plane of the sample, where X is perpendicular to the length of the leadframe leg and Y is parallel to the length of the leadframe leg. The length of the whisker is parallel to the X-axis, as shown in Fig. 5. The gray scale in the map corresponds to the angle between the normal to the (3 2 1) plane and the Z-axis. Therefore, Fig. 5 shows the distribution of the angle between the normal vector of the (3 2 1) plane and the laboratory Z-axis. The reason for choosing the (3 2 1) normal for mapping is because we have found that the Sn grains in the finish have (3 2 1) texture. We shall discuss the texture later. In addition to this map, we obtained maps that show the angle between the normal of the (2 1 1) plane and Z-axis, between the normal of the (1 0 0) plane and Z-axis, etc. From the orientation information, the direction cosine between the normal vector of the crystal orientation plane (*abc*) and the laboratory axis [*XYZ*] can be obtained. This relationship is shown in Fig. 6 for the whisker, and the growth direction of this whisker is determined to be [0 0 1] and it is parallel to the X-axis in Fig. 5. The [0 0 1] growth direction of Sn whisker has also been confirmed by cross-sectional transmission electron diffraction and microscopy analyses [15].

In Fig. 5, the angle between the normal of the (3 2 1) plane and Z-axis of most grains is found to be very similar and is close to zero. This means that the surface of the solder finish has a texture of (3 2 1). This is confirmed by the pole figures of

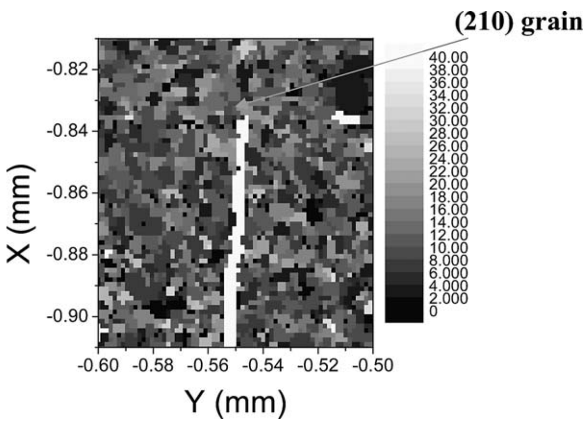

Fig. 5. Grain orientation of a whisker and surface grains of leadframe. It shows the distribution of the angle between the normal vector (3 2 1) of the grains and the laboratory Z-axis.

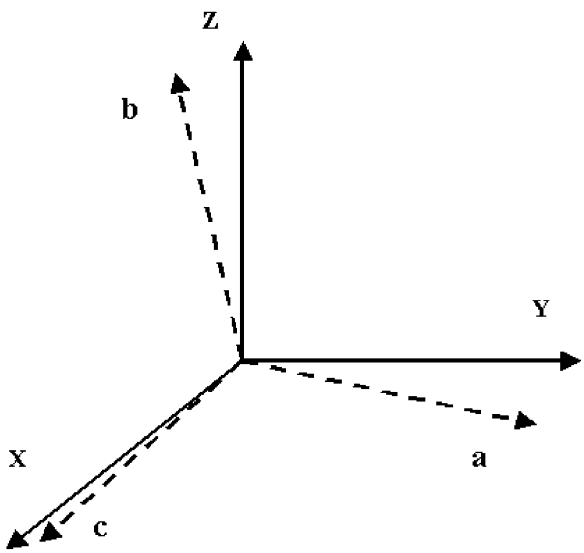

Fig. 6. Schematic diagram of relationship between laboratory axes (*XYZ*) and whisker crystal axes (*abc*).

grains in the finish. The pole figures of the normal of (1 0 0), (1 1 0), (3 2 1) and (2 1 1) planes are shown in Fig. 7(a)–(d), respectively. A high concentration of (3 2 1) at the center of the pole can be seen in Fig. 7(c), with a spreading of the poles about 10° from the center. This shows that the surface of the finish has the texture of (3 2 1) grains. But among the textured grains, there is a discontinuity in the grain orientation at the root of the whisker. As discussed above, the grain orientation of the specific grain just below the whisker has (2 1 0) orientation, rather than (3 2 1).

3.2. Stress analysis

The stress distribution on the solder finish around the whisker is shown in Fig. 8. Starting from Fig. 5, if we remove the X-ray data of the whisker itself by a software program to allow the stress around the whisker root to be analyzed, we obtain Fig. 8, which has slightly larger grids than those in Fig. 5. Physically, it means that we have polished the whisker away, yet the root of the whisker is left in the finish. We note that the coordinates of the root of the whisker are $(x,y) = (-0.8415, -0.5475)$ in both Fig. 5 and Fig. 8, as

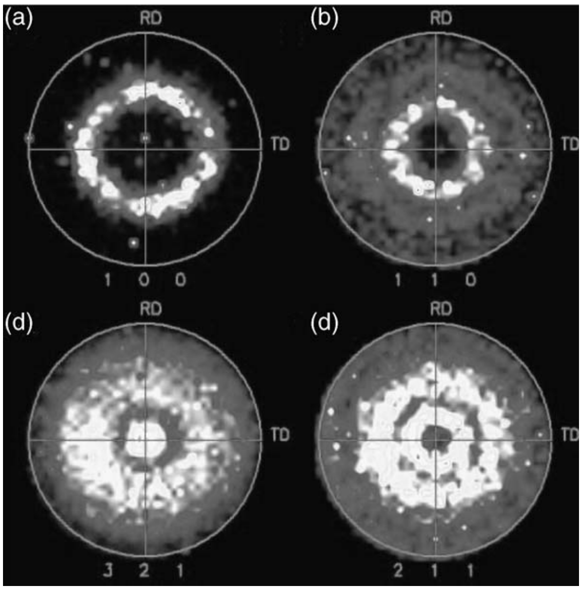

Fig. 7. The pole figures of grains in the CuSn finish. (a) (1 0 0) pole, (b) (1 1 0) pole, (c) (3 2 1) pole, and (d) (2 1 1) pole.

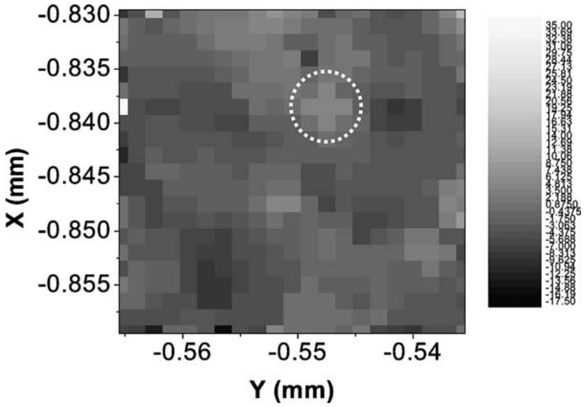

Fig. 8. Deviatoric stress along the *Z*-direction (equivalent to the biaxial stress) around the whisker root. In the analysis, the diffraction contribution of the whisker has been removed.

indicated by a circle in Fig. 8. Fig. 5 shows the grain orientation distribution, and Fig. 8 shows the corresponding average biaxial compressive stress distribution. In the latter, a light-colored cross (within the circle) can be seen at the root of the whisker, where $(x,y) = (-0.8415, -0.5475)$. Our X-ray micro-diffraction study shows that at a local area of 100 μm × 100 μm, the stress is highly inhomogeneous, with local variations from grain to grain. The finish is therefore under biaxial stress only on average. This is because each whisker has relaxed the stress in the region surrounding it. But, the stress gradient around the root of a whisker does not have radial symmetry. What Fig. 8 shows is a plot of $-\sigma'_{zz}$, which is the deviatoric component of the stress along the surface normal. Since, $\sigma'_{xx} + \sigma'_{yy} + \sigma'_{zz} = 0$ by definition, $-\sigma'_{zz}$ is a measure of the in-plane stress (note that for a blanket film, with free or passivated surface, on average the total normal stress $\sigma_{zz} = 0$), from which σ_b (biaxial stress) $= (\sigma_{xx} + \sigma_{yy})/2 = (\sigma'_{xx} + \sigma'_{yy})/2 - \sigma'_{zz} = -3\sigma'_{zz}/2$. This relation is always true on average. A positive value of σ'_{zz} indicates an overall tensile stress, whereas a negative value indicates an overall compressive stress. The absolute value of stress (about 4 MPa) in the whisker root (the light-colored cross) is higher than that in the surrounding grains. If we assume the whisker to be stress-free, the surface of SnCu finish is under compressive stress. However, the stress values, corresponding to a strain of less than 0.01%, are only slightly larger than the strain/stress sensitivity of the white beam Laue technique (sensitivity of the technique is 0.005% strain). No very long range stress gradient has been observed around the root of a whisker, indicating that the growth of a whisker has only relaxed most of the local compressive stress to a distance of several grains in the surrounding.

The numerical value and the distribution of stress are shown in Table 1, where the root of the whisker is again at the same "$x = -0.8415$" and "$y = -0.5475$" coordinates, as in Figs. 5 and 8. Overall, the compressive stress is quite low, of the order of several MPa; however, we can still see the slight stress gradient (about 1 MPa/μm) going from the whisker root area to the surroundings. It means the stress level just below the whisker is slightly less compressive than that in the surrounding area. This is because the stress near the whisker has been relaxed by whisker growth. In Table 1, the long and dark-colored arrows indicate the directions of stress gradient. In the table, some blocks or grids next to each other show a similar stress level, which might mean that they belong to the same grain.

4. Discussion

For spontaneous Sn whisker growth, there are three necessary and sufficient conditions. The first is the kinetics of fast atomic diffusion at room temperature. The second is the driving force of compressive stress. The third is a protective surface oxide needed for the stress gradient and the localized growth of whiskers. The first one is given since Sn has a low melting point of 232 °C, so the self grain boundary diffusion in Sn at room temperature is fast; the grain boundary diffusivity is about 10^{-8} cm^2/s [12]. Also, Cu diffuses interstitially in Sn, and room temperature formation of Cu_6Sn_5 occurs between Cu and Sn [10]. We measured the stress distribution in the finish. We shall discuss below the source of the stress and its distribution in relationship to the second and the third conditions.

Fig. 9 depicts the cross-section of a Sn whisker on a bi-layer of Sn (or eutectic SnCu) and Cu. The cross-section is cut along the length of the whisker depicted in Fig. 4 and is viewed along the *Y*-axis. There exists a layer of Cu_6Sn_5 between the Sn and Cu and a surface oxide layer on the SnCu finish. Both the Cu_6Sn_5 and the surface oxide have been

Table 1
Deviatoric stress distribution around whisker

1.5 μm

(Unit : Mpa)

	-0.5400	-0.5415	-0.5430	-0.5445	-0.5460	-0.5475	-0.5490	-0.5505	-0.5520	-0.5535	-0.5550
-0.8340	-2.82	-3.21	-2.26	0.93	0.93	-0.23	-8.17	2.22	1.49	1.6	-0.03
-0.8355	-2.26	-2.64	-2.64	-1.04	1.37	1.37	-1.31	0.87	0.87	0.87	-0.7
-0.8370	-2.53	-3.21	-3.21	-2.64	-1.04	3.61	0.75	0.87	0.7	0.7	-0.19
-0.8385	-7.37	-9.62	-6.57	-2.64	3.61	4.52	3.61	0.29	-1.31	0	-4.79
-0.8400	-7.37	-8.22	-6.57	-1.18	0.75	4.23	0.75	-2.25	-2.27	-2.91	-6.91
-0.8415	-4.17	-4.84	-4.17	-1.81	-0.67	0.00	-1.96	-1.96	-3.74	-5.08	-5.08
-0.8430	-4.17	-4.17	-3.63	-1.81	-1.81	-2.29	-2.29	-1.96	-1.96	-3.27	-3.27
-0.8445	-4.14	-4.17	-3.86	-3.63	-2.79	-4.64	-4.78	-0.84	-1.4	-1.49	-3.27
-0.8460	-3.14	-3.63	-3.86	-3.63	-3.13	-4.78	-4.78	0.04	0.04	-1.41	-2.33
-0.8475	-4.14	-4.49	-4.49	-4.64	-3.86	-6.04	-1.72	3.55	3.55	-0.41	-2.33
-0.8490	-3.33	-5.67	-6.29	-6.29	-2.66	-2.08	-1.72	-1.79	0	-1.79	-3.73

Whisker

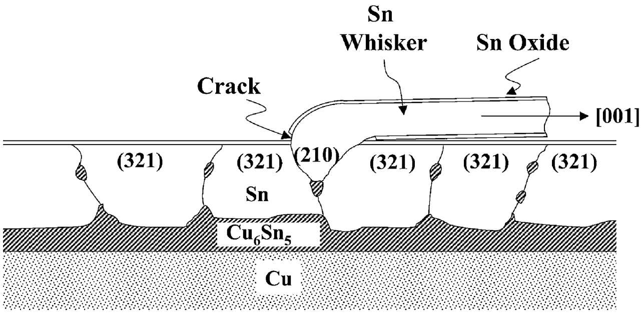

Fig. 9. Schematic diagram of a cross-section depicting the orientation of the whisker and surface grains in the SnCu finish, the Cu_6Sn_5 layer between the Cu leadframe and SnCu finish, and the grain boundary precipitates of Cu_6Sn_5. The whisker is almost parallel to the surface of the finish. Both have surface oxide, and there is a crack in the oxide at the root of the whisker.

observed experimentally [15]. The matrix of Sn contains many precipitates of Cu_6Sn_5 in the grain boundaries of Sn [15]. The specific grain under the whisker is shown to have a [2 1 0] orientation, which is different from the surrounding grains with a [3 2 1] texture. The surface of the whisker is shown to have an oxide layer, which has been observed too [16]. It is expected since the whisker growth occurs in ambient atmosphere. However, the oxide at the root of the whisker is assumed to have a crack. Without the crack, the whisker is completely confined in oxide; it cannot grow. Therefore, the growth of the whisker must cause a crack in the oxide and the crack is the entrance for vacancies.

We have measured the compressive stress in the SnCu solder finish on Cu leadframe by X-ray micro-diffraction. Where does this compressive stress come from? If we consider the coefficient of thermal expansion (CTE) of Sn and Cu, the CTE of Sn (26.7 ppm/°C) is higher than that of Cu (16.6 ppm/°C); hence, the Sn (or SnCu) layer should be under tension at room temperature after a soldering cycle. Also, if a leadframe is bent, one side of the leadframe is in tension and the opposite side is in compression, yet we found whiskers on both sides. Therefore, we must conclude that the compressive stress that leads to spontaneous Sn whisker growth is due to neither thermal stress nor mechanical stress. Rather, it is due to the room temperature chemical reaction between Sn and Cu to form Cu_6Sn_5 IMC. The chemical energy per atom to form the compound is about 0.5 eV/atom, which is four to five orders of magnitude larger than the strain energy per atom at the elastic limit of Sn, about 10^{-5} eV/atom. So, chemical reaction can occur and produce strain in the sample; the trade-off is favorable. In the SnCu solder finish on Cu leadframe, we have found that the Cu_6Sn_5 forms as precipitate in the grain boundaries of the finish [15]. The Cu atoms from the leadrame diffuse into the finish to lead to growth of the grain boundary IMC. The volume increase of IMC growth will exert a compressive stress on the grains on both sides. If we consider a given volume "V" in the Sn finish that contains an IMC precipitate, the diffusion of a Cu atom into this volume to react with Sn to grow on the IMC will produce a stress,

$$\sigma = -B\frac{\Omega}{V} \tag{1}$$

where σ is stress, B is bulk modulus, and Ω is partial molecular volume of a Cu atom in Cu_6Sn_5 (we ignore the molar volume change of Sn atoms in the reaction for simplicity). The negative sign indicates that the stress is compressive. When more and more Cu atoms, say "n" Cu atoms, diffuse into the volume V to form Cu_6Sn_5, the stress in Eq. (1) increases by changing Ω to $n\Omega$. To relieve the stress, some Sn atoms will diffuse out from the volume V to the root of a whisker to enlarge the stress-free whisker.

In other words, if we consider the eutectic SnCu layer in Fig. 9 as an open system, the inward diffusion of Cu atoms driven by IMC formation produces a compressive stress in the layer. To relieve the stress, it requires the outward diffusion (laterally) of Sn atoms to the root of a whisker to lengthen the stress-free whisker in order to compensate for the volume increase caused by the influx of Cu atoms. The stress just below the root of the whisker is close to zero, which means that the stress is almost completely relaxed by the whisker growth, but there is a stress gradient in the surrounding of the root and the stress increased gradually with distance from the root. This stress gradient drives the lateral diffusion of Sn. What is unclear yet is the atomic mechanism of incorporation of Sn atoms at the root of a whisker.

We also need to consider how to break the surface oxide locally to grow the whisker. Intuitively, it seems that the microstructure of the Sn grain at these broken spots could be different from that of their surrounding grains. In other words, it is a structural discontinuity, where the surface oxide can be broken easily. From the pole figures in Fig. 7, we found that the surface of the solder finish has a texture of (3 2 1). In Fig. 8, the grain (part of the whisker) just below the whisker showed (2 1 0) orientation. This grain is a discontinuity in the textured matrix. The compressive stress may be able to break the surface oxide along the grain boundaries between this grain and its surrounding grains. Therefore, such a grain may become the seed of a whisker. The broken oxide or the crack at the root of the whisker should be kept open for the supply of vacancies needed for the lateral diffusion of Sn atoms.

The oxide on the whisker surface has two effects; it reduces the surface energy and it defines the diameter of the whisker. The latter is due to the fact that the gain in strain energy is spent in creating the surface of the whisker. By balancing the strain energy against the surface energy in a unit length of the whisker, $\pi R^2\varepsilon = 2\pi R\gamma$, we find

$$R = \frac{2\gamma}{\varepsilon} \tag{2}$$

where R is radius of the whisker, γ is surface energy per unit area, and ϵ is strain energy per unit volume. Since strain energy at the elastic limit per atom (about 10^{-5} eV/atom) is about four to five orders of magnitude smaller than chemical bond energy or surface energy per atom of the oxide (about 0.5 eV/atom) , the radius or diameter of a whisker is found to be several micrometers, which is about four orders of magnitude larger than the atomic diameter of Sn.

Very often, we observed bent whiskers as shown in Ref. [15]. One of them is sketched in Fig. 9. Since the stress gradient may be inhomogeneous in radial directions around the root of a whisker, and if on one side of the root the stress gradient is smaller than on the other sides, the growth rate there will be slower. We assume that when it is slow enough to be less than a certain rate, the oxide crack may have time to heal itself by oxide growth in ambient atmosphere. This is indicated on the right-hand side of the root of the whisker depicted in Fig. 9. If this happens, whisker growth will stop on this side, resulting in a bending of the whisker when the opposite side continues to grow.

5. Summary

We have studied the orientation and stress of Sn whiskers and the surrounding grains at their roots by X-ray micro-diffraction with synchrotron radiation. On the eutectic SnCu solder finishing surface, we found long Sn whiskers, and the growth direction is [0 0 1]. There is a compressive stress in the SnCu solder finish, and the stress level is less than 10 MPa. Around the root of a whisker, there are also stress gradients extending out only over a few grains, and the stress just below the whisker is zero. The SnCu solder finishing surface has a texture of (3 2 1) grains, but the grain (part of the whisker) just below the whisker showed (2 1 0) orientation.

Acknowledgements

The authors at UCLA would like to acknowledge the support from SRC/NSC Contract No. NJ-853. The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, Materials Science Division, of the US Department of Energy under Contract No. DE-AC03-76SF00098 at Lawrence Berkeley National Laboratory.

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